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Evaluation Of Vapor Pressure And Ultra-High Vacuum Tribological Properties Of Ionic Liquids (2) Mixtures and Additives

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ABSTRACT

Ionic liquids are salts, many of which are typically viscous fluids at room temperature. The fluids are characterized by negligible vapor pressures under ambient conditions. These properties have led us to study the effectiveness of ionic liquids containing both organic cations and anions for use as space lubricants. In the previous paper we have measured the vapor pressure and some tribological properties of two distinct ionic liquids under simulated space conditions. In this paper we will present vapor pressure measurements for two new ionic liquids and friction coefficient data for boundary lubrication conditions in a spiral orbit tribometer using stainless steel tribocouples. In addition we present the first tribological data on mixed ionic liquids and an ionic liquid additive. Post mortem infrared and Raman analysis of the balls and races indicates the major degradation pathway for these two organic ionic liquids is similar to those of other carbon based lubricants, i.e. deterioration of the organic structure into amorphous graphitic carbon. The coefficients of friction and lifetimes of these lubricants are comparable to or exceed these properties for several commonly used space oils.

INTRODUCTION

The first Apollo missions including a vehicle (lunar roving vehicle, LRV) to transport the astronauts (Apollo 15 in 1971 and Apollo 17 in 1972) had specifications for operation in an environment of $\pm 120^{\circ}$ C for as much as 2 years. In order to expedite the delivery schedule, these requirements were eventually reduced to operation in 0-100°C for only a few

hours. In order to provide adequate lubrication to the wheel drive unit (traction drive assembly) with lubricants available at the time, the units were sealed with a nitrogen atmosphere at approximately $\frac{1}{2}$ atm. (5x10⁴ Pa) pressure. Two separate reports list the lubricant for the traction drive assembly as KrytoxTM 143AZ oil [1] or KrytoxTM 143AC oil [2], both of which are oils used for space lubrication. Other lubricating materials included KrytoxTM 240AC grease in the traction drive and steering motors as well as silicone base oil in the suspension damper. Dry lubrication was also employed. In 2004, President George W. Bush set forth a Vision for Space Exploration Program in which we would return to the moon. The specifications for lubrication of rovers and other moving mechanical assemblies include a five year life time during which the lubricant may have to endure the temperature extremes of the lunar diurnal cycle (-170 to 110°C at equator). Current liquid lubricants do not have attractive vapor pressures at the peak temperatures and become too viscous at the lower temperature extremes making them undesirable. Furthermore, future missions will involve longer lunar traverses than the few miles driven during Apollo 15 and Apollo 17 [3] in what is now known to be an abrasive environment created by the fine, electrically charged or magnetic particulate lunar regolith. Planned exploration may include traverses into totally shaded craters where ice may reside, imposing a lubricant requirement of operation at a temperature of -233°C.

For these reasons we have undertaken a search for more suitable base oils to be used for liquid lubricants on the moon and mars. Ionic liquids (ILs) have been demonstrated to have exceptionally high boiling points, remarkably low freezing points and extremely low vapor pressures compared to terrestrial liquid lubricants. They have also been shown in the literature to have eutectic properties further lowering the freezing point. Little work appears in the literature for either the application of mixed fluids for space lubrication or for their use as additives for other ionic liquid base oils. This paper serves to bridge the gap between the prior work [4] evaluating pure ionic liquids as potential space lubricants and the ability to enhance the property of the base ionic liquid for use in space applications. In addition, new vapor pressure data will be presented to extend the scant vapor pressure database.

EXPERIMENTAL

Materials

Two new commercially available ionic fluids, referred to henceforth as IL-C and IL-D respectively (to prevent confusion with IL-A and IL-B used in our prior presentation [4]), were used as received for vapor pressure determinations. Dilute solutions (1 μ g/mL) of both ILs, their 1:1 mixture (henceforth referred to is IL-C+D) and the mixture with a solid IL additive (1 wt %) were prepared in dichloromethane for syringe loading onto ball bearings for spiral orbit tribometer (SOT) testing [5, 6].

SOT specimens were made of standard AISI 440C stainless steel cleaned by polishing with 0.05 μm silicon carbide to a roughness of 0.05 μm and sonicated twice in water immediately prior to use. Between 25-50 μg of dilute IL solution was delivered to the balls held magnetically on a rotating fixture. The lubricant load was determined by weighing.

Instrumentation

The SOT is illustrated in Figure 1 and is described in the literature [5, 6]. The SOT simulates an angular contact bearing by employing a single ½ inch ball rolled between a fixed and rotating plate at 30 rpm. The load applied in these experiments provided a mean Hertzian stress of 1.5G pa. For vacuum tests, the pressure at the start of all experiments was $\leq 2 \times 10^{-8}$ Torr (2.6 $\times 10^{-6}$ Pa). The coefficient of friction (CoF) was recorded as a function of ball orbits and the life time was determined as the orbit number when the friction coefficient rose to 0.2. Unless otherwise specified, the normalized lifetimes reported, defined as the number of orbits at failure divided by the amount of lubricant in μ g, are the average of at least 3 trials.

Vapor pressure (VP) measurements were obtained using a modified Knudsen cell technique [7]. Vapor pressures were determined at several elevated temperatures and a plot of lnVP vs. 1/T was constructed with T in K. Room temperature VP were determined by extrapolation to 20°C (293 K).

Infra red spectra (IR) were acquired on a Fourier transform spectrometer. The spectrometer was equipped with a 3 inch diameter integrating sphere and mount as described in the literature [8] for acquiring spectra from the balls and a microscope with 32 times objective for examination of the wear tracks and scrub regions of the disc and guide plates. The microscope provided a 100 μ m field of view. The balls, guide plates and discs were further subjected to Raman microspectrometric analysis which typically provided a 14 μ m spot diameter. The Raman spectrometer used the 514.5 nm argon ion line and cooled CCD detector to facilitate data collection.

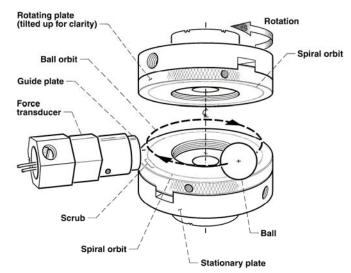


Figure 1. Schematic of an UHV spiral orbit tribometer.

RESULTS

Vapor pressures are to be compared to those of typical space lubricants abstracted from the literature [9] and those of other ILs [4] reported in Table 1.

| Fluid | VP (Pa at 20°C) | |
|---------------------------------|-------------------------|--|
| Krytox™ 143AB | 2.0x10 ⁻⁴ | |
| Fomblin™ Z-25 | 3.9x10 ⁻¹⁰ | |
| Pennzane [®] SHF X2000 | 0 2.2x10 ⁻¹¹ | |
| IL-A | 2.9x10 ⁻⁹ | |
| IL-B | 1.3x10 ⁻¹² | |

Table 1. Vapor Pressures of select space lubricants and ILs.

The Spiral Orbit Tribometer operates at low speed and under high load to accelerate testing. This combination of load, speed and small amount of lubricant ($< 50~\mu g$) allowed the system to operate in the boundary lubrication regime. The ball was rolling and pivoting in a spiral and maintained in the orbit by the guide plate. The force the ball exerted on the guide plate was used to determine the friction coefficient, since the ball was sliding between the disks at this moment. As the lubricant

was tribologically stressed, it was degraded and eventually consumed. Test conclusion was defined when a friction coefficient of 0.28 was attained, Figure 2. Normalized lubricant lifetime (or inversely, its degradation rate) is reported. For example, the data in Figure 2 for 25 μg of IL-C yields a normalized lifetime of 488 orbits per μg . The results for the new ILs, their mixture and their mixture with the additive IL are to be compared to the existing IL database, Table 2.

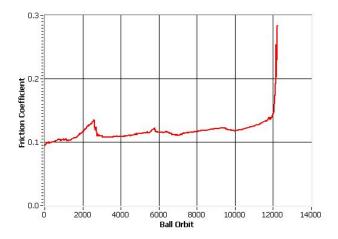


Figure 2. SOT trace for 25 μg of IL-C.

| Fluid | CoF | Lifetime (orbit/ug) |
|----------------|------|------------------------|
| Krytox 143AB | 0.14 | 43 |
| Fomblin 815Z | 0.12 | 44 |
| Pennzane 2001A | 0.08 | 4110 |
| IL-A | 0.08 | 1843 |
| IL-B | 0.09 | 1627 |
| IL-C | 0.10 | 337 |
| IL-C+D | 0.11 | 507 |

Table 2. SOT lifetime and friction coefficient data for space lubricants and ILs run in ultra-high vacuum.

Post mortem analysis of balls, guide plates and discs yielded information about the degradation mechanism for the ILs. The IR and Raman spectra showed patterns similar to other carbon based lubricants for the wear areas indicating the degradation mechanism to be consistent with those of other carbon based liquid lubricants tested under these conditions [10].

DISCUSSION

The vapor pressures to be reported for these new ILs indicate their potential for use as space lubricants. These fluids have vapor pressures superior to the some of the best space lubricants currently available although they may still not be

good enough for application in the lunar environment of 10^{-14} Torr $(1.3x10^{-12} \text{ Pa})$ especially when considering the estimated five year lifetime requirement.

These ILs, their mixture and mixture with additive have reasonable CoF for the commonly used bearing material they were tested against. The lifetimes are also superior to some of the currently used space lubricants mentioned in Table 2. With the possibility to synthesize an estimated 10¹⁸ potential ILs, there are plenty of other candidates to consider.

REMARKS

- 1) The ILs have vapor pressures superior to some currently employed space lubricants.
- 2) ILs, their mixtures and IL additive mixtures possess adequately low friction coefficients for use as lubricants.
- 3) The lifetimes of the newly reported ILs, their mixtures and IL additive mixtures could use some improvement.

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